

USSR/Organic Chemistry - Synthetic Organic Chemistry, E-2

Abst Journal: Referat Zhur - Khimiya, No 19, 1956, 61542

Abstract: transacylation as a result of which is formed benzyl-urethane (X) and 2-aminoquinuclidine; the latter split off NH_3 and is converted to VIII. On heating of IV with IX (or with $\text{C}_6\text{H}_5\text{NH}_2$) to 180° NH_3 is evolved and together with VIII there is formed respectively di-benzyl-(XI) or diphenyl-(XII)-urea. XI is formed also on heating IX with X to 180° . A solution of 3.76 g II in 40 ml absolute alcohol is mixed with 7.27 ml 16.4% alcoholic solution of HCl, there are added within 20 minutes with cooling with ice and stirring 3.88 g III, mixing is continued for 3 hours at $\sim 20^\circ$, then the mixture is boiled for 4 hours, evaporated in vacuum and the residue is treated with 50% solution of K_2CO_3 . Extracted with ether; the residue after evaporation of ether is heated 30 minutes in boiling water bath and ground with dry ether; yield of IV is 44.3%, MP $166-168^\circ$; hydrochloride MP $136-138^\circ$ (from aqueous acetone). From mother liquor isolated 1 g VI, BP $87-89^\circ/0.5$ mm, $122-123^\circ/14$ mm, n_D^{23} 1.4723, hydrochloride MP 300° (decomposition). Under analogous conditions were prepared V (BP $105-108^\circ/0.6$ mm, n_D^{16} 1.4587) and VII (n_D^{16} 1.4671). Mixture of 1 g IV and 10 ml HCl (1:1) boiled for 4 hours filtered, evaporated in vacuum, acetone is added,

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USSR/Organic Chemistry - Synthetic Organic Chemistry, E-2

Abst Journal: Referat Zhur - Khimiya, No-19, 1956, 61542

Abstract: NH_4Cl , filtrate evaporated, residue treated with 50% solution
 K_2CO_3 and extracted with CHCl_3 ; thus VIII is obtained; picrate
MP 158-160°.

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CIA-RDP86-00513R001445830002-4



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2/ 5/ 4 Pyridylglutaric acid and products of its transforma-

2

"APPROVED FOR RELEASE: 08/22/2000

CIA-RDP86-00513R001445830002-4

PM 8/22

APPROVED FOR RELEASE: 08/22/2000

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RUB + SOV, M.V.

10.000 Ha 1.0 Benz. Chloride
III was hydrolyzed with 14
294 + 2 M.E.

mye

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CIA-RDP86-00513R001445830002-4

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RUBTSOV, M.V.

E-2

USSR/ Organic Chemistry - Synthetic organic chemistry

Abs Jour : Referat Zhur - Khimiya, No 4, 1957, 11738

Author : Rubtsov M.V., Mikhlin Ye.Ye., Furshtatova V.Ya.
Title : Preparation of Isonicotinic Acid

Orig Pub : Zh. prokl. khimii, 1956, 29, No 6, 946-948

Abstract : A method has been developed for the preparation of isonicotinic acid (I) by oxidation with dilute HNO_3 of the mixture of γ -methylolpicolines (II) formed on heating mixture of β - and γ -picolines (III, IV) with formalin (V) at atmospheric pressure. It is shown that in lieu of HNO_3 a mixture of HNO_3 and H_2SO_4 can be successfully utilized in the oxidation. An experimental study is made of the preparation of I from citric acid (VI); a more precise determination has been made of the conditions of preparation, with increased yields, of 2,6-dihydroxy isonicotinic acid (VII) and 2,6-dichlor isonicotinic acid (VIII); yields of I have been considerably increased. 117.6 g technical mixture III and IV (the mixture contains 15% water and 40% IV, on the dry basis) and 200 g V are boiled 15 hours; III and excess V are steam distilled, aqueous solution of II is concentrated to 160-180 ml and

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Abs Jour : Referat Zhur - Khimiya, No 4, 1957, 11738

these are added, within 20 minutes, to 350 ml of 57.5% HNO_3 heated to 98° , heating is continued for 4 hours, after which neutralization is effected with 65-75 g Na_2CO_3 to obtain I, yield 77.5-85% (on basis of IV), MP 314° . From distillate, by addition of 32 g KOH and 48 g NaCl, are isolated 48-56 g III. Trimethyl ester of VI, 73 g, is shaken for 15-20 minutes with 730 ml 25% aqueous solution of NH_3 , the mixture is evaporated in vacuum, 365 g of 73% H_2SO_4 are added, the mixture is slowly heated to 125° , and held at $125-130^\circ$.

Card 2/2

RUBTSOV, M.V.; NIKITSKAYA, Ye.S.

Preparation of N-ethylpiperidine. Zhur. prikl. khim. 29 no.12:1887
D.'56. (MLBA 10r6)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S. Ordzhonikidze.
(Piperidine)

RUBISOV, M V

455

AUTHORS: Yakhontov, L. N., and Rubtsov, M. V.

TITLE: Synthesis of Quinuclidone-2

PERIODICAL: Zhurnal Obshchey Khimii, 1957, Vol. 27, No. 1, pp. 72-77 (U.S.S.R.)

ABSTRACT: Using ethyl ether of pyridyl-4-acetic acid, the authors synthesized a second oxo-derivative - quinuclidone-2 - which is bicyclic amide. The Arndt-Euster method giving a 37% yield of $\text{CH}_2\text{COOC}_2\text{H}_5$ from isonicotinic acid was found to be the most suitable for this type of reaction. Hydrogenation with a platinum catalyst prepared according to Adams gave a considerable yield of ethyl ether of piperidyl-4-acetic acid. Saponification of the latter gave chlorohydrate of piperidyl-4-acetic acid which, by means of thionyl chloride, was converted into homologous acid chloride. By subjecting the latter to reaction with calcined potash in anhydrous chloroform, it converted into quinuclidone-2, an oily substance which together with hydroxylamine forms a crystalline oxime. The derivation of quinuclidone-2 oxime from ethyl ether of quinuclidine-carboxylic acid-2 is described. There are 7 non-Slavic references.

Card 1/2

455

Synthesis of Quinuclidone-2

ASSOCIATION: The All-Union Scientific-Research Chemical-Pharmaceutical Institute
im. S. Ordzhonikidze (Vsesoyuznyy Nauchno-Issledovatel'skiy Khimiko-
Farmatsevticheskiy Institut im. S. Ordzhonikidze)

PRESENTED BY:

SUBMITTED January 30, 1956

AVAILABLE:

Card 2/2

KURTSOV, M V

456

AUTHORS: Mikhlina, Ye. Ye., and Rubtsov, M. V.

TITLE: Synthesis of 3-Methylquinuclidine-2-carboxylic Acid (Sintez 3-metilkhinuklidinkarbonovoy kisloty)

PERIODICAL: Zhurnal Obshchey Khimii, 1957, Vol. 27, No. 1, pp. 77-83 (U.S.S.R.)

ABSTRACT: The synthesis of di-substituted quinuclidine derivatives - 3-methylquinuclidine-2-carboxylic acid from gamma-ethylpyridine, is described. Condensation of the gamma-ethylpyridine with dioxymalonic ester and consequent conversion of 1,1-dicarboethoxy-2-(pyridyl-4')-propene-1 into 3-methylquinuclidine-2-carboxylic acid was considered the most simple synthesis method. However, instead of 1,1-dicarboethoxy-2-(pyridyl-4')-propene-1, ethyl ether alpha-oxy-alpha carboethoxy-beta-(pyridyl-4)-butyric acid was obtained. Reduction of the diester in the presence of platinum oxide and consequent saponification and decarboxylation led to alpha-oxy-beta-(piperidyl-4)-butyric acid. In order to convert the latter compound into 3-methylquinuclidine-2-carboxylic acid, it was necessary to substitute the alpha-oxy group in the acid with a haloid. The acid was treated with thionyl chloride. At a temperature of 60-65°, only acid chloride was formed; increased temperatures to 70-75° resulted in intensive resinification of

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Synthesis of 3-Methylquinuclidine-2-carboxylic Acid

the substance, and no change of the alpha-oxy group into Cl group was observed.

This result indicates that the alpha-oxy group in the acid is less active, as a result of which the synthesis of 3-methylquinuclidine-2-carboxylic acid from alpha-oxy-beta-(piperidyl-4)-butyric acid has proven impossible.

There are 5 references, of which 3 are Slavic.

ASSOCIATION: All-Union Scientific-Research Chemical-Pharmaceutical Institute im. S. Ordzhonikidze (Vsesoyuznyy Nauchno-Issledovatel'skiy Khimiko-Farmatseyticheskiy Institut im. S. Ordzhonikidze).

PRESENTED BY:

SUBMITTED: January 30, 1956

AVAILABLE:

Card 2/2

RUBEN M.

U.S. DEPARTMENT OF JUSTICE
FEDERAL BUREAU OF INVESTIGATION
WASHINGTON, D.C. 20535

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CIA-RDP86-00513R001445830002-4"

A-4 Sci Res Chem Pharm. Inst. 1958, Vol. 28, No. 1, pp. 103-110 (USSR)

AUTHORS: Mikhlin, Ye. Ye. , Rubtsov, M. V. 79-1-22/63
 TITLE: The Synthesis of Quinuclidine-3-Acetic Acid (Sintez khinuklidin-3-uksusnoy kisloty)
 PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol.28, Nr 1, pp.103-110(USSR)

ABSTRACT: The present paper describes the synthesis of quinuclidine-3-acetic acid which makes it possible to transfer the investigation to the 3-substituted derivatives of quinuclidine. 4-(β -oxyethyl) was first used as initial product for this synthesis with the attempt of synthesizing 1,1,1-trichloro-2-oxy-3-(pyridyl-4')-4-oxybutane from this product by reaction with chloral. This attempt failed, as only resin-like products were obtained. The tests with 4-(β -acetoxyethyl)-pyridine (83%) obtained from 4-(β -oxyethyl)-pyridine (formula I) only yielded 4-vinylpyridine as final product. 4-(β -methoxyethyl)-pyridine (VI) proved to be a more stable compound during the influence of chloral. The condensation of this product with chloral in the presence of acetic piperidine (see the reaction scheme) led to 1,1,1-trichloro-2-oxy-3-(pyridyl-4')-4-oxybutane.

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79-1-22/63

The Synthesis of Quinuclidine-3-Acetic Acid

-3-(pyridyl-4')-4-methoxybutane (VII), where no substituted 4-vinylpyridine was produced (as it was the case in the analogous reaction of 4-(β -acetoxyethyl)-pyridine with chloral). The compound (VII) was by reaction with potassium alcoholate converted to 4-methoxy-3-(pyridyl-4')-crotonic acid (VIII). Its ethyl ester (IX) was at room temperature and in the presence of a platinum catalyst converted to the ethyl ester of methoxy-3-(piperidyl-4')-butyric acid (X). In order to exchange the methoxy group for halide and for the purpose of a synthesis of the derivative of quinuclidine the compound (X) was heated at 100 - 120°C with 67 % hydrobromic acid in a tube soldered shut. 4-bromo-3-(piperidyl-4')-butyric acid synthesized in this connection was esterified and in the presence of pyridine subjected to cyclization. The ethyl ester of quinuclidine-3-acetic acid (XI) finally resulted, which was after saponification converted to quinuclidine-3-acetic acid. There are 8 references, 4 of which are Slavic.

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79-1-22/63

The Synthesis of Quinuclidine-3-Acetic Acid

ASSOCIATION: All-Union Scientific Chemical-Pharmaceutical Research Institute imeni S. Ordzhonikidze
(Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut im. S. Ordzhonikidze)

SUBMITTED: January 7, 1957

AVAILABLE: Library of Congress

Card 3/3

1. Chemistry 2. Quinuclidine-Synthesis 3. Acetic acid

AUTHORS: Furshtatova, V. Ya., Mikhlina, Ye. Ye., 79-28-3-23/61
Rubtsov, M. V.

TITLE: The Synthesis of the 6-Carboxymethyl-1-Diazocyclo-(3,2,1)-
octane-7-Carboxylic Acid and Some of its Derivatives
(Sintez 6-karboksimetil-1-azabitsiklo-(3,2,1)-oktan-7-
karbonovoy kisloty i nekotorykh yeye proizvodnykh)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 3, pp. 668-675
(USSR)

ABSTRACT: A number of works is dealing with the synthesis and the
biological investigation of the derivatives of quinuclidine,
the 1 - diazocyclo - (2,2,2) - octane (refs.1-3). The
dicyclic system isomeric to quinuclidine, the 1-diazocyclo-
(3,2,1)octane, has however, not been sufficiently
investigated until now. Only a limited amount of
6 - monosubstituted 1 - diazocyclo-(3,2,1) -octanes were
obtained. The substituted octanes of the mentioned structure
were not synthesized. Among the 2,3-disubstituted compounds
of quinuclidine synthesized by the authors a number of
biologically active products was found so that it was also

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The Synthesis of the 6-Carboxymethyl-1-Diazocyclo-octane-7-Carboxylic Acid and Some of its Derivatives

79-28 3-23/61

to
of interest/obtain the isomeric 6,7-disubstituted 1-Diazocyclo-(3,2,1) octanes and to compare the biological and chemical properties of the compounds of two isomeric series with each other. In the present work the synthesis of 6-carboxymethyl - 1 -diazocyclo-(3,2,1)-octane-7-carboxylic acid and some derivatives is described. It was carried out according to the mentioned scheme (see formulae (I) to (X)). Thus the synthesis of 6-carboxymethyl-1-diazocyclo - (3,2,1) octane-7-carboxylic acid is described. The reaction process is shown as follows: From the ethylester of β -(pyridyl-3)-acrylic acid passing through the ethylesters of β -dicarboxymethyl- β -(piperidyl - 3)-propionic acid, β -carbethoxybromoethyl-(piperidyl -3)-propionic acid to the diethylester of 6-carboxymethyl-1-diazo-(3,2,1)-octane- 7,7 -dicarboxylic acid. Together with these mentioned products the following compounds are synthesized: 1.- The diethylester of 6-carboxymethyl-1-diazocyclo-(3,2,1)-octane-7-carboxylic acid.
2.- The di(diethylaminoethyl)- and di-(dimethylaminoethyl) ester of the 6-carboxymethyl-diazocyclo-(3,2,1)-octane-7-carboxylic acid. 3. 6-(β -oxymethyl)-7-Oxymethyl)-diocyclo-

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The Synthesis of the 6-Carboxymethyl-1-Diazocyclo-
octane-7-Carbocyclic Acid and Some of its Derivatives

79-28.3-23/61

(3,2,1)-octane and 6 (β -chloroethyl)-7-chloromethyl-1-
diazocyclo-(3,2,1)-octane.

There are 4 references, 2 of which are Soviet.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-
farmatsevticheskiy institut imeni S. Ordzhonikidze (All-
Union Scientific Chemical and Pharmaceutical Research
Institute imeni S. Ordzhonikidze

SUBMITTED: March 16, 1957

Card 3/3

AUTHORS: Furshtatova, V. Ya., Mikhlina, Ye. Ye., 79-28-5-8/69
Rubtsov, M. V.

TITLE: Synthesis of 6,7-Di-substituted 1-Azabicyclo-
-(3,2,1)-Octane (Sintez 6,7-dizameshchennykh 1-azabitsiklo-
-(3,2,1)-oktana)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 5,
pp. 1170-1176 (USSR)

ABSTRACT: In the last publication by the authors (Reference 1) a
simple synthesis of 6-carboxymethyl-1-azabicyclo-(3,2,1)-
octane-7-carboxylic acid and of its derivatives was descri-
bed. Most interesting of these compounds were the proper-
ties of the ethyl esters of 6-carbethoxymethyl-1-azabicyclo-
-(3,2,1)-octane-7-carboxylic acid. Thus this ester hydro-
lyzed easily in aqueous solution under formation of an aci-
dous ester. The same way also reacts the isomeric ethyl ester
of 3-carbethoxymethylquinuclidine-2-carboxylic acid which
converts to 3-carbethoxymethylquinuclidine on the same
conditions. The comparison of the two isomeric diesters
makes possible the assumption, that the saponification of

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Synthesis of 6,7-Di-substituted 1-Azabicyclo- 79-28-5-8/69
-(3,2,1)-Octane

of the carbethoxyl group in ethyl ester of the 6-carbethoxymethyl-1-azabicyclo-(3,2,1)-octane-7-carboxylic acid (in the mentioned scheme) takes place in position 7 and that the acidous ester forming on this occasion has the structure (II) of the scheme. From this a whole number of 7-alkyl-(aryl)-aminoethyl-6-(β -oxyethyl)-1-azabicyclo-octanes and of esters of 7-dialkylaminoethyl-6-(β -oxyethyl)-1-azabicyclo-(3,2,1)-octane were obtained. The compound (II) converts to compound (III) by means of thionylchloride; this compound was further treated with alkyl-(aryl)-amines. The amides (IV) obtained then were reduced to the compound (V) by means of lithium aluminum hydride. On treating this with chlorine anhydrides of some acids the corresponding esters (VI) resulted. On the conversion of (V) with thionylchloride the compound (VII) was obtained in which the chlorine atom in the 6- β -chloroethyl group is of limited activity as experiments showed. There are 2 Soviet references.

Card 2/3

Synthesis of 6,7-Di-substituted 1-Azabicyclo- 79-28-5-8/69
-(3,2,1)-Octane

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S. Ordzhonikidze (All-Union Scientific Chemical and Pharmaceutical Research Institute imeni S. Ordzhonikidze)

SUBMITTED: May 25, 1957

Card 3/3

AUTHORS: Yakhontov, L. N., Yatsenko, S. V., 79-28-5-9/69
Rubtsov, M. V.

TITLE: Synthesis of Substituted Quinuclidyl-2-Carbinol
 (Sintez zameshchennykh khinuklidil-2-karbinolov)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 5,
 pp. 1177-1181 (USSR)

ABSTRACT: P. Rabe, in 1911 was the first to realize the synthesis of the substituted quinuclidyl-2-carbinols of the quinine-alkaloidal type (Reference 1). This method consists of the condensation of the ethylesters of β [N-benzoyl-piperidyl-(4)]-proprionic acid and any other acid (e. g. cinchoninic acid or quininic acid) with subsequent closing of the quinuclidine cycle, and by reduction of the obtained ketone with the corresponding substituted quinuclidyl-2-carbinol resulting as final product (see scheme 1). Until our time this scheme was the only one for the synthesis of substituted quinuclidyl-2-carbinols. According to this scheme quinine (Reference 2), hydroquinine (Reference 3) as well as a series of analogs and isomers

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Synthesis of Substituted Quinuclidyl-2-Carbinol 79-28-5-9/69

of quinine alkaloids (References 4-6) were synthesized. In the present work another method for the synthesis of substituted quinuclidyl-2-carbinols is described (see scheme 2). As initial product serves 2-formylquinuclidine (Reference 7) which in the conversion with different organomagnesium compounds forms the corresponding substituents of quinuclidyl-2-carbinol. This way the following carbinols were synthesized: (quinuclidyl-2)-methylcarbinol (I), (quinuclidyl-2)-ethylcarbinol (II) and (quinuclidyl-2)-(naphthyl-1-)-carbinol (III). The compound (I) was also obtained by reduction of the 2-acetylquinuclidine (Reference 8) (IV) in the presence of a platinum catalyst (scheme 3), on which occasion also a mixture of diastereoisomeric (quinuclidyl-2)-methylcarbinols formed in crystalline and oily state.

There are 8 references, 5 of which are Soviet.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S. Ordzhonikidze (All-Union Scientific Chemical and Pharmaceutical Research Institute imeni Ordzhonikidze)

SUBMITTED: April 15, 1957

Card 2/2

AUTHORS: Chizhov, A. K., Rubtsov, M. V.

79-28-5-10/69

TITLE: Synthesis of 1-Azabicyclo-(3,2,1)-Octane-7-Carboxylic Acid
(Sintez 1-azabitsiklo-(3,2,1)-oktan-7-karbonovoy kisloty)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 5,
pp. 1181 - 1183 (USSR)

ABSTRACT: Only the synthesis of the unsubstituted bicycle and some of its 6-substituted cycles are reported with respect to the 1-azabicyclo-(3,2,1)-octanes (References 1-3). The synthesis of unknown 1-azabicyclo-(3,2,1)-octanes with substituents in position 7 as far as these are isomeric to the 2-substituents of quinclidine, i.e. of 1-azabicyclo-(2,2,2)-octane, are of great interest. To these belong the quinine alkaloids and a number of other products obtained in the last years. Among them compounds with valuable biological properties were discovered. The present work has as its purpose the synthesis of the 1-azabicyclo-(3,2,1)-octane-7-carboxylic acid which again can serve as initial basis for the synthesis of the 7-substituted 1-azabicyclo-(3,2,1)-octane. The synthesis of this acid can be realized according to the mentioned scheme (formulae I to VII):

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79-28-5-10/69

Synthesis of Azabicyclo-(3,2,1)-Octane-7-Carboxylic Acid

The nicotine aldehyde (II) was condensed with malonic acid ester in the presence of piperidine at room temperature; there the diethyl ester of 2-(pyridil-3')-2-oxyethane dicarboxylic acid of the acid-1,1(II) formed. The heating of the latter (II) with acetic anhydride caused the splitting off of a molecule of water and the separation of diethyl ester of 2-(pyridil-3')-vinyl dicarboxylic acid-1,1 (III). The chlorine hydrate of (III) was reduced in alcohol solution in the presence of platinum oxide according to Adams, the chlorine hydrate of diethyl ester of the 2-(piperidil-3')-ethanedicarboxylic acid-1,1 (IV) having been obtained on this occasion. In order to further make possible the conversion from 3-substituted piperidine to the azabicyclic system, compound (IV) was treated with bromine in chloroform. The synthesized diethyl ester of 2-(piperidil-3')-1-bromethanedicarboxylic acid-1,1 (V) on heating with pyridine converted to 7,7 dicarboethoxy-1-azabicyclo-(3,2,1)-octane (VI). On boiling this diester with concentrated hydrochloric acid the chlorine hydrate of 1-azabicyclo-(3,2,1)-octane-7-carboxylic

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79-28-5-10/69

Synthesis of 1-Azabicyclo-(3,2,1)-Octane-7-Carboxylic Acid

acid (VII) resulted. There are 3 references, 1 of which is Soviet.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut (All-Union Scientific Chemical and Pharmaceutical Research Institute)

SUBMITTED: April 15, 1957

Card 3/3

AUTHORS: Yakhontov, L.N., Rubtsov, M.V. SOV/79-28-11-45/55

TITLE: Reduction of the Harmine Derivatives to the Derivatives of the
Pyridine Tetrahydro Harmine With Sodium Boro-Hydride (NaBH_4)
(Vosstanovleniye borgidridom natriya proizvodnykh garmina v proiz-
vodnyye Py-tetragidrogarmina)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 11, pp 3108-3112 (USSR)

ABSTRACT: The investigation of various methods of transforming the harmine
derivatives by reduction to the Py-tetrahydro harmine derivatives
caused the authors to conclude that the best reducing agent among
those hitherto suggested in these methods is the sodium boro-hydride.
It was shown that only the quaternary salts of harmine are reduced.
The harmine itself and its non-quaternary derivatives do not react
with NaBH_4 . Therefore, in the cases where the derivatives of Py-tetra-
hydro harmine are not substituted at the Py-nitrogen the Py-N-chloro-
benzylate of harmine is reduced with a subsequent removal of the
benzyl group by the hydration of the Py-N-benzyl tetrahydro harmine
on a palladium catalyst. This Py-N-chloro benzylate of harmine was
obtained in a yield of 95 % by heating equimolecular amounts of
harmine and benzyl chloride in benzyl alcohol at 120° within 12 hours.

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SOV/79-28-11-45/55

Reduction of the Harmine Derivatives to the Derivatives of the Pyridine Tetrahydro Harmine With Sodium Boro-Hydride (NaBH_4)

The reduction of Py-N-chloro benzylate of the harmine takes place with methyl alcohol by gradual addition of sodium boro-hydride (duration 3 hours). The yield of the hydrochloride of Py-N-benzyl tetrahydro harmine amounted to 90 %. The Py-N-benzyl tetrahydro harmine was also obtained in another way: By the reduction of harmine with sodium alcoholate according to Fischer (Fisher-Ref 1) to the Py-tetrahydro harmine, which then was subjected to the benzylation by benzyl chloride with potash at 110-120°. The final product (as hydrochloride) (78 %) was identical with the previous. Both compounds had the same constants, the same solubility, the same results of the analyses, as well as the same ultraviolet spectra (Figure). The debenylation of the Py-N-benzyl tetrahydro harmine obtained with sodium boro-hydride by the hydration on palladium also yielded Py-tetrahydro harmine, which was identical with that obtained by the reduction of harmine with sodium alcoholate (Scheme). Similar results were also obtained in the experiments with norharmine derivatives (Scheme 2).--There are 1 figure and 3 references, 1 of which is Soviet.

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SOV/79-28-11-45/55

Reduction of the Harmine Derivatives to the Derivatives of the Pyridine Tetrahydro Harmine With Sodium Boro-Hydride (NaBH_4)

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S.Ordzhonikidze (All-Union Scientific Chemo-Pharmaceutical Research Institute imeni S.Ordzhonikidze)

SUBMITTED: October 3, 1957

Card 3/3

AUTHORS: Yakhontov.L.N., Yatsenko,S.V., Rubtsov,M.V. SOV/79-28-11-47/55

TITLE: Synthesis of 4-Aminopiperidine (Sintez 4-aminopiperidina)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 11, pp 3115-3119 (USSR)

ABSTRACT: The 4-aminopiperidine is a semiproduct for the production of biologically active compounds. According to reference 1 some N-substituted 4-aminopiperidines have spasmolytic activity (Ref 1). There is, however, no convenient synthesis of this compound mentioned in publications. Its two described syntheses by the reduction from 4-aminopyridine and from acyclic compounds give only small yields. In this paper a convenient preparative synthesis of the dichloro hydrate of 4-aminopiperidine from isonicotinic acid in two steps with a yield of 66 % is described. In its elaboration various ways of synthesizing the 4-aminopiperidine from isonicotinic acid were checked, which is now used as industrial raw material (Scheme). The reactions by Hofmann, Curtius, and Schmidt (Gofman, Kurtsius, Shmidt) were used for the transformation of the carboxyl group. According to the first method the isonicotinic acid according to reference 4 was converted by way of the ester into the amide and further on according to Hofmann into the aminopiperidine. Basing on the second method the isonicotinic acid was converted into hydrazide according to reference 6 by way of the

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Synthesis of 4-Aminopiperidine

SOV/79-28-11-47/55

ester. This was reduced by way of platinum to the hydrazide of the isonipecotic acid, which according to Curtius was converted to the 4-aminopiperidine. The synthesis by the reduction of the isonicotinic acid to the isonipecotic acid with subsequent substitution of the carboxyl group by the amino group according to Schmidt turned out to be the most convenient method. The Schmidt reaction takes place best with sodium azide in the presence of H_2SO_4 , as it is convenient in preparative respect and is not connected with a previous development of poisonous vapours of hydrazoic acids (yield 66 %) as is the case in using hydrazoic acid. In the checking of the first method according to Hofmann the catalytic reduction of the aminopyridine to the 4-aminopiperidine was realized.- There are 8 references, 1 of which is Soviet.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S.Ordzhonikidze (All-Union Scientific Chemo-Pharmaceutical Research Institute imeni S.Ordzhonikidze)

Card 2/3

RUBTSOV, M.V., prof., otv. red.; PERSHIN, G.N., prof., zam. otv. red.;
MAGIDSON, O.Yu., prof., red.; MASHKOVSKIY, M.D., prof., red.;
UTKIN, L.M., prof., red.; RUZHENTSEVA, A.K., prof., red.;
SHCHUKINA, M.N., prof., red.; BAYCHIKOV, A.G., kand. tekhn. nauk,
red.; MIKHALEV, V.A., kand. khim. nauk, red.; RYAZANTSEV, M.D.,
kand. tekhn. nauk, red.; SUVOROV, N.N., kand. khim. nauk, red.;
PLYASHKEVICH, A.M., st. nauchnyy sotr., red.

[Basic trends in the work of the S.Ordzhonikidze All-Union Chemico-pharmaceutical Scientific Research Institute; survey of its activity from 1920 to 1957] Osnovnye napravleniia rabot VNIKhFI; obzor deiatel'nosti za 1920-1957 gg. Moskva, 1959. 649 p. (MIRA 15:5)

1. Moscow. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut.

(CHEMISTRY, MEDICAL AND PHARMACEUTICAL)

AUTHORS: Mikhlina, Ye. Ye., Rubtsov, M. SOV/79-29-1-27/74

TITLE: Cyano-Ethylation of Quinuclidone-3 (Tsianetilirovaniye khinuklidona-3)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 1, pp 118-124 (USSR)

ABSTRACT: A very interesting problem is represented by the cyano-ethylation of quinuclidone-3 hitherto not investigated. The present paper deals with this question. In its transformation with an excess of acrylonitrile into dioxane or tertiary butyl alcohol in the presence of 30 % potash lye in methyl alcohol, a mixture of mono- and dicyano-ethylated products is formed. The general yield of mono- and dicyano-ethylated quinuclidones, as well as their quantitative relation obtained depends on the solvent used. The yield of them thus amounts to about 44 % if the reaction is performed in dioxane. The main product of the mixture (85 %) is represented in this case by the dicyano-ethylated quinuclidone-3. The substitution of tertiary butyl alcohol for dioxane increases the total yield up to 70 %, while at the same time also the percentage of monocyano-ethylated quinuclidone-3 increases (about 35 % of the total sum of cyano-ethylated products). On a reaction of quinuclidone-3

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Cyano-Ethylation of Quinuclidone-3

SOV/79-29-1-27/74

with acrylonitrile at the molar ratio the yield of these products is 10 % only. On the basis of the reactions performed the structure of formula (II) was assigned to the mono-cyano-ethylated quinuclidone-3. The reduction of quinuclidone (IV) yielded the quinuclidine (VI). The synthesis performed is presented by scheme 1. The dicyano-ethylated quinuclidone-3 can have the structure 2,2- or 2,4-di-(β -ethyl cyanide)-quinuclidone-3. By saponification of the ketonitrile the keto-diacid is formed. The latter is not transformed into the tri-cyclic unsaturated compound (A) on heating with acetic acid anhydride, which would be the case if the ethyl cyanide groups were in position 2 and 4. On the basis of these data, the structure (VII) is the only correct one for the dicyano-ethylated quinuclidone-3. The quinuclidines (XIII), (IX), and (X) the dihydrazide (XI) and further quinuclidines (XII), (XIII), and (XIV) were synthesized from it according to scheme 2. There are 4 references, 3 of which are Soviet.

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Cyano-Ethylation of Quinuclidone-3

SOV/79-29-1-27/74

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S. Ordzhonikidze (All-Union Chemico-pharmaceutical Scientific Research Institute imeni S. Ordzhonikidze)

SUBMITTED: November 30, 1957

Card 3/3

AUTHORS: Chizhov, A. K. , Rubtsov, M. V. SOV/79-29-1-29/74

TITLE: Synthesis of the 7-Monosubstituted Compounds of 1-Azabicyclo-
-[3,2,1]-Octane (Sintez 7-monozameshchennykh 1-azabitsiklo-
-[3,2,1]-oktanov)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 1, pp 130-136 (USSR)

ABSTRACT: In the previous report (Ref 1) the authors described the
synthesis of the 1-azabicyclo-[3,2,1]-octane-7-carboxylic acid.
Owing to its reactive carboxyl group this acid can be used in
the synthesis of various 7-monosubstituted compounds of the
1-azabicyclo-[3,2,1]-octanes. These compounds are very inter-
esting in the biological research work as far as among the
isomeric 2-monosubstituted compounds of quinuclidine (Ref 2)
pharmacologically active products could be found. In the -
present paper the synthesis of the 7-monosubstituted compounds
of 1-azabicyclo-[3,2,1]-octanes were described, i.e. of the
amides, amines, hydrazides, esters, alcohols, halides, chloric
acid anhydrides of the acids and some quaternary salts. For the
synthesis of these compounds the 1-azabicyclo-[3,2,1]-octane-
-7-carboxylic acid (I) was used as initial product which was
transformed into the chloric acid anhydride (II) and furthermore

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Synthesis of the 7-Monosubstituted Compounds of
1-Azabicyclo-[3,2,1]-Octane

SOV/79-29-1-29/74

into the 7-monosubstituted compounds of 1-azabicyclo-[3,2,1]-octanes according to the scheme mentioned. The synthesized bases (VI), (XI), (XVIII), and (XX) were converted by methyl iodide into the corresponding methiodides (VII), (XII), (XIX), and (XXI). The dimethiodides of the 7-(γ -diethyl-amino propyl)-1-azabicyclo-[3,2,1]-octane (XIX) and diethyl-amino ethyl ester of the initial acid (XXI) show a pronounced blocking action on the ganglia of the vegetative nerve system which as regards its character approaches the effect of dioquine, the dimethiodide of the diethyl-amino ethyl ester of the quinuclidine-2-carboxylic acid (Ref 4). It was shown that the halogen atom in the molecule of 7-chloro methyl-1-azabicyclo-[3,2,1]-octane, unlike the 2-chloro methyl quinuclidine described in publications, has a high mobility and is able to undergo condensation with the sodium malonic ester. There are 4 references, 3 of which are Soviet.

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Synthesis of the 7-Monosubstituted Compounds of
1-Azabicyclo- 3,2,1 -Octane

SOV/79-29-1-29/74

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cheskiy institut imeni S. Ordzhonikidze (All-Union Chemico-
pharmaceutical Scientific Research Institute imeni
S. Ordzhonikidze)

SUBMITTED: November 14, 1957

Card 3/3

SOV/79-29-2-25/71

AUTHORS: Nikitskaya, Ye. S., Usovskaya, V. S., Rubtsov, M. V.

TITLE: Piperidine Derivatives as Possible Hypotensive Agents (Proizvodnyye piperidina kak vozmozhnyye gipotensivnyye sredstva)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 2, pp 472-476 (USSR)

ABSTRACT: According to the sec tertiary amines of the quinuclidine and piperidine series, which develop a high ganglion-blocking activity, the authors synthesized some N-substituted piperidine derivatives, in order to examine further tertiary amines. 2,6-lutidine, a waste product in the preparation of "phthivazide" (Ftivazid), served as initial product. The reaction of 2,6-lupetidine (obtained from 2,6-lutidine) with the chloric anhydride of β -chloropropionic acid and subsequent boiling of the reaction product in ethyl alcohol with piperidine and diethyl amine gave the compounds (I) and (II). By reduction, the latter correspondingly passed over to compounds (III) and (IV) (Scheme). After a number of failures, the authors succeeded in carrying out the synthesis, beginning from 2,6-lupetidine, of the sec quaternary salts by the aid of dichloric anhydride of glutaric and adipic acid, namely, compounds (V) and (VI). These

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Piperidine Derivatives as Possible Hypotensive Agents

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piperidides of both acids could, correspondingly, be converted by reduction into 1,5-bis(2',6'-dimethyl piperidine-1')-pentane (VII) and 1,6-bis(2',6'-dimethyl piperidine-1')-hexane (VIII). Sec quaternary salts (Scheme 2) easily result from these two compounds. By reaction of ethyl ester of 6-methyl pipecolic acid with chloric anhydride of β -chloro propionic acid and by subsequent treatment of the reaction product with piperidine or diethyl amine, piperidines (IX and X) were obtained, which in their turn changed over to piperidines (XI and XII) by reduction (Scheme 3). The constants of the compounds synthesized will be given in a following paper. There is 1 Soviet reference.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S. Ordzhonikidze (All-Union Scientific Chemical-pharmaceutical Research Institute imeni S. Ordzhonikidze)

SUBMITTED: January 3, 1958

Card 2/2

AUTHORS: Furshtatova, V. Ya., Mikhlina, Ye. Ye., SOV/79-29-2-26/71
Rubtsov, M. V.

TITLE: Investigation of the Formation Reaction of N-Substituted
2-Aminomethyl-3-Vinyl Quinuclidines (Izucheniye reaktsii
obrazovaniya N-zameshchennykh 2-aminometil-3-vinilkhinukli-
dinov)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 2, pp 477-485 (USSR)

ABSTRACT: The question is raised in the present paper, whether the
N-substituted compounds of 2-aminomethyl-3-(β -oxyethyl)-
quinuclidine can be transformed into N-substituted compounds
of 2-aminomethyl-3-vinyl quinuclidine by distilling the re-
spective stearates and benzoates at normal pressure. Esters
were obtained by the reaction of chloric anhydride of stearic
and benzoic acid with the N-substituted compounds of
2-aminomethyl-3-(β -oxyethyl)-quinuclidine in benzene solution.
On distilling quinuclidine (I) two quinuclidines (II and III)
were formed. They were separated by treating the mixture with
mercury acetate in acetic acid solution, involving the sub-
sequent separation of the product of the affiliation of
mercury acetate to the unsaturated compound (II) and the

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Investigation of the Formation Reaction of
N-Substituted 2-Aminomethyl-3-Vinyl Quinuclidines

SOV/79-29-2-26/71

separation of (II). Besides (II) and (III) also ethyl stearate was separated. The formation of compound (II) is evidently accompanied by a separation of stearic acid (Scheme 1). Only the tricyclic derivative (III) and ethyl benzoate (Scheme 2) result from the distillation of compound (IV). A similar process is observed on heating quinuclidine (V) up to boiling temperature, in which connection benzoic acid, besides (III) is separated (Scheme 3). Heating of the compounds (VI) and (IX) with phthalic anhydride in the presence of benzene sulfo acid at 285° led only to compound (III) (Scheme 4). The structure of 2,3-(3',4'-N-ethyl piperidine)-quinuclidine was proven by a counter-synthesis, proceeding from 3-carbethoxy methyl quinuclidine-2-carboxylic acid. There are 5 references, 2 of which are Soviet.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S. Ordzhonikidze (All-Union Scientific Chemico-pharmaceutical Research Institute imeni S. Ordzhonikidze)

SUBMITTED: January 3, 1958
Card 2/2

AUTHORS: Yanina, A. D., Rubtsov, M. V. SOV/79-29-2-27/71

TITLE: The Hofmann Cleavage of 1-Azabicyclo-(3,2,1)-Octane (Gofman-ovskoye rasshchepleniye 1-azabitsiklo-(3,2,1)oktana)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 2, pp 485-493 (USSR)

ABSTRACT: In the Hofmann cleavage (Refs 1,2) the unsymmetric bicyclic systems react with a common nitrogen, under formation of not one monocyclic heterocycle but 3 heterocycles which have an unsaturated side chain or under the formation of their transformation products, in dependence on the question, from which β -carbon atom the hydrogen will separate to form the water molecule. The direction of cleavage apparently depends both on the stability of the bicycles and on experimental conditions. This was proven by the experimental cleavage in the case of 1-azabicyclo-(3,2,1)-octane (III). The initial product in this connection was 1-carbethoxy methyl-3-carbethoxy piperidine (I) (Scheme 1). The intramolecular condensation of (I) into (II) proceeded in the presence of potassium alcoholate [not with metallic potassium (Ref 3)], which increased the yield from 30 to 71 %. By reduction of the ketone (II) octane (III) was obtained, which was transformed by methyl iodide into the

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The Hofmann Cleavage of 1-Azabicyclo-(3,2,1)-Octane SOV/79-29-2-27/71

quaternary salt (IV) and further with silver oxide into the corresponding base (V). It was found that on cleaving 1-azabicyclo-(3,2,1)-octane under various conditions (at normal pressure, in vacuum, in 40 % potash lye, and at increased pressure) three products are formed: 1-methyl-3-allyl pyrrolidine, 1-methyl-3-(β -oxyethyl)-piperidine, and di-[β -(1-methyl piperidyl-3)]-ethyl ether. 1-methyl-3-allyl pyrrolidine was separated in all cases and is the chief product of the cleavage of 1-azabicyclo-(3,2,1)-octane in alkali medium, in vacuum, and at normal pressure. On its heating in water under pressure, 1-methyl-3-(β -oxyethyl)-piperidine is the chief product. There are 8 references, 1 of which is Soviet.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S. Ordzhonikidze (All-Union Scientific Chemico-pharmaceutical Research Institute imeni S. Ordzhonikidze)

SUBMITTED: January 3, 1958

Card 2/2

5(3)

SOV/79-29-4-35/77

AUTHORS:

Yakhontov, L. N., Rubtsov, M. V.

TITLE:

Synthesis of the Derivatives of Py-N-benzyltetrahydronorgarmine-3-carboxylic Acid (Sintez proizvodnykh Py-N-benziltetragidronorgarmin-3-karbonovoy kisloty)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 4, pp 1201-1206 (USSR)

ABSTRACT:

The esters and amides of the above acid are of importance as intermediate products for the synthesis of reserpine analogues; however, no description has as yet been given because of the difficulties encountered in preparing them (except in reference 1). The general method developed by the authors at an earlier date for the reduction of "garmine" derivatives by means of sodium boron hydride resulting in the Py-tetrahydrogarmine derivatives rendered possible the preparation of the ethyl ester (XII) and N-methylanilide (X) of Py-N-benzyltetrahydronorgarmine-3-carboxylic acid (Scheme), starting from Py-N-chlorobenzylate of norgarmine-3-carboxylic acid (VII) or its betaine (VI) via the ethyl ester (XI) and N-methylanilide (VIII). Because of the difficulties encountered the previous synthesis of the above chlorobenzylate (Ref 3) was replaced by the following method: Com-

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Synthesis of the Derivatives of Py-N-benzyltetrahydronorgarmine-3-carboxylic Acid

pound (IV) was obtained from garmine (I) by two alternative methods; either garmine was changed into (III) by reaction with benzaldehyde, and (III) was heated for 13 hours at 160° with benzylchloride in a benzyl alcohol medium; or, in the second process, "garmine" was transformed, with benzyl chloride, into compound (II) which was slightly heated with benzaldehyde in the presence of pyridine. The quantities of the Py-N-chlorobenzylate of 3-styrylnorgarmine obtained amounted to 46 and 58.8%, respectively (details in the experimental part). During the oxidation of the Py-N-chlorobenzylate of 3-styrylnorgarmine with potassium permanganate the betaine of Py-N-benzylnorgarmine-3-carboxylic acid forms. It was suggested generally to synthesize the amides of norgarmine-3-carboxylic acid by the reaction with amines and phosphorus oxychloride at 160-170°. There are 3 references, 2 of which are Soviet.

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Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S.Ordzhonikidze (All-Union Scientific Chemico-pharmaceutical Research Institute imeni S. Ordzhonikidze)

Synthesis of the Derivatives of Py-N-benzyltetrahydronorgarmine-3-carboxylic
Acid

SOV/79-29-4-35/77

SUBMITTED: March 21, 1958

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5(3)

SOV/79-29-6-38/72

AUTHORS: Furshtatova, V. Ya., Mikhlina, Ye. Ye., Rubtsov, M. V.

TITLE: Synthesis of the Substituted Compounds of the 7-Aminomethyl-6-(β -aminoethyl)-1-azabicyclo-(3,2,1)-octane (Sintez zameshchennykh 7-aminometil-6-(β -aminoetil)-1-azabitsiklo-(3,2,1)-oktana)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 6, pp 1945 - 1949 (USSR)

ABSTRACT: For the purpose of carrying out the synthesis of the 6,7-diaminosubstituted compounds of 1-azabicyclo-(3,2,1)-octane the hydrochloride of 6-carboxymethyl-1-azabicyclo-(3,2,1)-octane-7-carboxylic acid (I) was converted into the corresponding acid chloride (II) by means of thionyl chloride. The latter was reacted with alkyl (aryl) amines and the amides (III) were obtained. The reduction of the amides with aluminum-lithium hydride led to the substituted compounds of the 7-aminomethyl-6-(β -aminoethyl)-1-azabicyclo-(3,2,1)-octane (IV) (Scheme 1). In the investigation of the properties of the diamines synthesized (IV) it was found that diamines which contain a non-substituted hydrogen atom bound to nitrogen, may be converted into the tricyclic system 6,7-(3',4'-N'-alkyl piperidino)-1-azabicyclo-

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Synthesis of the Substituted Compounds of the SOV/79-29-6-38/72
7-Aminomethyl-6-(β -aminoethyl)-1-azabicyclo-(3,2,1)-octane

(3,2,1)-octane (V) in the distillation in vacuum (Scheme 2). The formation of the tricyclic system (V) in this distillation was confirmed by the opposite synthesis of 6,7-(3',4'-N-benzyl piperidino)-1-azabicyclo-(3,2,1)-octane (V a) according to scheme 3. The 7-benzyl aminomethyl-6-(β -oxyethyl)-1-azabicyclo-(3,2,1)-octane (Ref 4) was converted into 7-benzyl aminomethyl-6-(β -chloroethyl)-1-azabicyclo-(3,2,1)-octane by means of thionyl chloride which yielded the compound (V a) in boiling with pyridine. There are 4 Soviet references.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S. Ordzhonikidze (All-Union Scientific Chemo-pharmaceutical Research Institute imeni S. Ordzhonikidze)

SUBMITTED: May 15, 1958

Card 2/2

GUSENKOV, P.V.; NATRADZE, A.G.; KORZHENEVSKIY, E.S.; RUETSOY, M.Y.; PERSHIN,
G.N.; MAGIDSON, O.Yu.; KRAFT, M.Ya.; YAKOVLEVA, Ye.V.; SMIRENSKIY, S.P.

M.D. Riazantsev; obituary. Med.prom. 14 no.2:64 F '60. (MIRA 13:5)

(RIAZANTSEV, MIKHAIL DMITRIEVICH, 1892-1960)

RUBTSOV, M.V.; MIKHLINA, Ye.Ye.; YAKHONTOV, L.N.

Chemistry of quinuclidine derivatives. Usp.khim. 29
no.1:74-105 Ja '60. (MIRA 13:6)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevti-
cheskiy institut imeni S. Ordzhonikidze.
(Quinuclidine)

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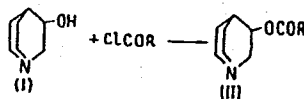
77374
SOV/79-30-1-35/78

AUTHORS: Mikhlin, Ye. Ye., Rubtsov, M. V.

TITLE: Synthesis of 3-Substituted Quinuclidine

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol 30, Nr 1, pp 163-171 (USSR)

ABSTRACT: Synthesis of several esters of 3-hydroxyquinuclidine is described. 3-Hydroxyquinuclidine (I) was obtained from 3-quinuclidone by reduction with lithium aluminum hydride in ether. Esterification of (I) was carried out with acid chlorides in benzene or in chloroform.



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Synthesis of 3-Substituted Quinuclidine

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3-(p-Aminobenzoyloxy)-quinuclidine was obtained by reduction of 3-(p-nitrobenzoyloxy)-quinuclidine over Raney nickel. The same reaction over Pt catalyst gave 3-(p-aminocyclohexanoyloxy)-quinuclidine. 3-(β -Phenylpropoxy)-quinuclidine was prepared by hydrogenation of 3-hydroxyquinuclidine ester and cinnamic acid. 3-(β -Cyanoethoxy)-quinuclidine (III), was obtained from 3-hydroxyquinuclidine and acrylonitrile in the presence of catalyst (30% KOH solution in methanol). 3-(γ -aminopropoxy)-quinuclidine (X) was formed by reduction of (III) with lithium aluminum hydride. Compound (III) was converted into 3-(β -carbethoxyethoxy)-quinuclidine (IV) in three different ways: (1) Nitrile (III) was heated with anhydrous alcohol and concentrated H_2SO_4 . (2) Nitrile (III) was hydrolyzed with subsequent esterification. (3) Dry HCl was bubbled through a boiling anhydrous alcohol solution of (III). The yield of (IV) was 60-75%. The best results were

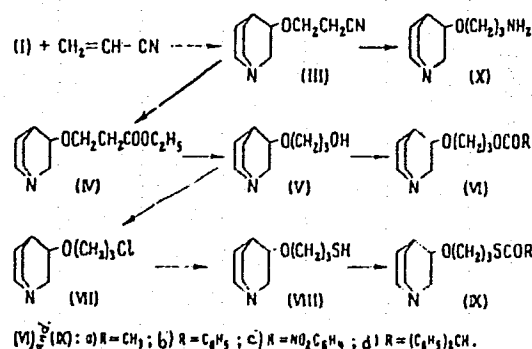
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Synthesis of 3-Substituted Quinuclidine

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obtained by the third method.



(IV) with lithium aluminum hydride is converted into (V), which on heating with alcohol and acid chlorides in benzene gave corresponding esters (VI). Thionyl chloride with (V) forms 3(γ -chloropropoxy)-quinuclidine

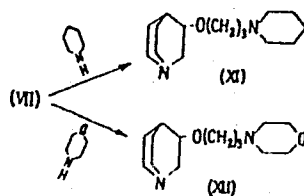
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Synthesis of 3-Substituted Quinuclidine

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(VII). The latter with thiourea and afterwards with alkali is converted into 3-(γ -mercaptopropoxy)-quinuclidine. Acid chlorides react with (VIII) forming thioesters. (VII) was heated with piperidine, morpholine, and diethylamine. In the first two cases corresponding 3-[γ -(N-piperidino)-propoxy]-quinuclidine (XI) and 3-[γ -(N-morpholino)-propoxy]-quinuclidine (XII) were obtained.



(VII) with diethylamine probably forms a polymeric compound of (VII). The pharmacological investigation was made by K. A. Zaytseva under the direction of M.

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Synthesis of 3-Substituted Quinuclidine

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SOV/79-30-1-35/78

D. Mashkovskiy. 3-Acetoxy-quinuclidine has strong cholinomimetic activity and 3-benzoyloxy-quinuclidine has hypotensive activity.

Table
Esters of 3-(γ -hydroxypropoxy)-quinuclidine

Nr	R	Yield (%)	bp (pressure in mm)	mp of hydrochloride	Empirical Formula
1	CH ₃	84	73—74° (0.4)	173—175°	C ₉ H ₁₅ O ₂ N · HCl
2	C ₂ H ₅	56.6	74—76 (0.3)	174—176	C ₁₀ H ₁₇ O ₂ N · HCl
3	C ₃ H ₇	58.2	84—85 (0.3)	175—177	C ₁₁ H ₁₉ O ₂ N · HCl
4	iso-C ₄ H ₉	72.5	88—90 (0.3)	180—182	C ₁₂ H ₂₁ O ₂ N · HCl
5	CH ₂ =CH-(CH ₂) ₈ *	73.7	227—230 (0.7)	—	C ₁₅ H ₂₇ O ₂ N
6	CH ₃ OCH ₂ *	65.5	101—104 (0.4)	172—174	C ₁₀ H ₁₇ O ₃ N
7	C ₂ H ₅ SCH ₂ *	77.7	118—119 (0.3)	—	C ₁₁ H ₁₉ O ₂ NS
8	C ₆ H ₅	71.5	148—150 (0.3)	238—240	C ₁₁ H ₁₇ O ₂ N · HCl

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Synthesis of 3-Substituted Quinuclidine

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Table cont'd

Nr	R	Yield (%)	bp (pressure in mm)	mp of hydrochloride	Empirical Formula
9	4-NO ₂ C ₆ H ₄	83.5	133-135 ***	256-258	C ₁₁ H ₁₆ O ₄ N ₂ · HCl
10	4-BrC ₆ H ₄	73.5	—	243-245 ****	C ₁₄ H ₁₈ O ₂ NBr · HCl · H ₂ O
11	4-ClC ₆ H ₄	88.5	—	198-200	C ₁₄ H ₁₈ O ₂ NCl · HCl
12	C ₆ H ₅ OCH ₂	89	180 (1)	165-167	C ₁₃ H ₁₉ O ₃ N · HCl
13	C ₆ H ₅ CH ₂ *	73.8	151-152 (0.3)	—	C ₁₅ H ₁₉ O ₂ N
14	C ₆ H ₅ CH=CH ₂	86.5	—	187-189	C ₁₆ H ₁₉ O ₂ N · HCl
15	3,4,5-(OCH ₃) ₃ C ₆ H ₂ *	44	67-70 ***	203-205	C ₁₇ H ₂₃ O ₅ N
16	3-C ₆ H ₄ N	71.2	141-142 (0.35)	231-233 *****	C ₁₃ H ₁₈ O ₂ N ₂ · 2HCl
17	4-C ₆ H ₄ N	50.2	149-150 (0.5)	238-240 *****	C ₁₃ H ₁₆ O ₂ N ₂ · 2HCl

* Empirical formula is given for the base.

*** mp of base.

**** Crystallized with 1 mole of H₂O.

***** mp is given for dihydrochloride.

Card 5/7

Synthesis of 3-Substituted Quinuclidine

77374

SOV/79-30-1-35/78

There is 1 table; and 5 references, 2 Soviet, 1 French, 2 U.S. The U.S. references are: L. H. Sternbach, S. Keiser, J. Am. Chem. Soc., 74, 2219 (1952); ibid. 74, 2215 (1952)

ASSOCIATION: Ordzhonikidze All-State Scientific-Research Chemical-Pharmaceutical Institute (Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S. Ordzhonikidze)

SUBMITTED: January 2, 1959

Card 7/7

RUBTSOV M.V.

5.3610

77375

SOV/79-30-1-36/78

AUTHORS: Nikitskaya, Ye. S., Usovskaya, V. S., Rubtsov,
M. V.

TITLE: Bicyclic Systems Based on 2,6-Lutidine. III.
N-Derivatives of 3-Oxa-9-azabicyclo-(3,3,1)-Nonane

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol 30, Nr 1, pp
171-182 (USSR)

ABSTRACT: Acyl and alkyl derivatives of 3-oxa-9-azabicyclo-(3,3,1)-nonane (I) were synthesized. Acid chlorides of acetic, propionic, and benzoic acids were reacted with I in anhydrous benzene with cooling and 9-acetyl- (IIa), 9-propionyl- (IIb), and benzoyl-3-oxa-9-aza-bicyclo-(3,3,1)-nonanes (IIc) were obtained. The obtained products, on reduction with lithium aluminum hydride, were converted into corresponding amines. Morpholine and dimethylamine in anhydrous alcohol, phenothiazine in anhydrous benzene, and the sodium salt of quinoxaline-4 in anhydrous alcohol were

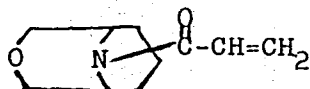
Card 1/10

Bicyclic Systems Based on 2,6-Lutidine. III

77375

SOV/79-30-1-36/78

reacted with 9-(β -chloropropionyl)-3-oxa-9-azabicyclo-(3,3,1)-nonane and corresponding β -substituted derivatives of 9-propionyl-3-oxa-9-azabicyclo-(3,3,1)-nonanes (IIId, IIe, IIIf, IIg) were obtained. The above reaction with phenothiazine and quinoxaline takes place with formation of a sideproduct, 9-acryloyl-3-oxa-9-azabicyclo-(3,3,1)-nonane.



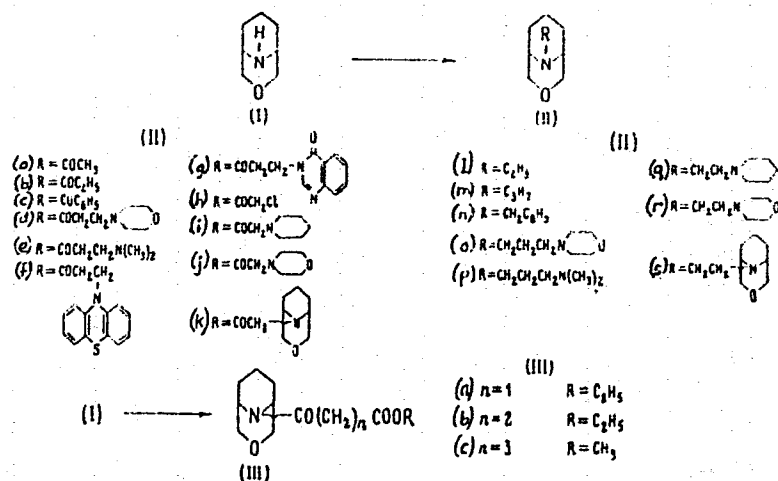
Acetyl chloride reacts with I, in aqueous alkali, forming as main product 9-[3'-oxa-9'-azabicyclo-3', 3', 1'-nonano-9']-acetyl-3-oxa-9-azabicyclo-(3,3,1)-nonane (IIj).

Card 2/10

Bicyclic Systems Based on 2,6-Lutidine. III

77375

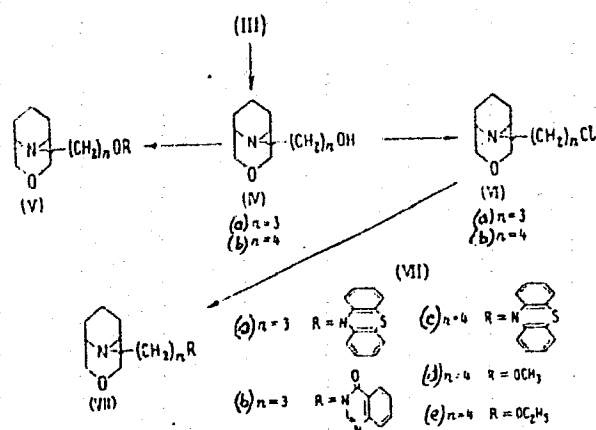
SOV/79-30-1-36/78



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Bicyclic Systems Based on 2,6-Lutidine. III

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SOV/79-30-1-36/78



Card 4/10

Bicyclic Systems Based on 2,6-Iutidine. III

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SOV/79-30-1-36/78

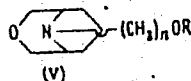
The corresponding amines (IIo, IIp, IIr, IIs, IIt) were obtained on reduction of IIId, IIe, III, IIj, IIk, with lithium aluminum hydride. Attempts to reduce compounds IIIf and IIg were unsuccessful. The desired amines were prepared as follows: I was reacted with carbethoxyacetyl chloride. The obtained IIIa was reduced to IVa; the latter with thionyl chloride gave VIa. Phenothiazine and quinoxalin-4-one were reacted with VIa; corresponding VIIa and VIIb were obtained. IIIb and IIIc were obtained similarly from β -carbethoxypropionyl chloride and β -carbomethoxypropionyl chloride, forming on reduction IVb. Thionyl chloride was reacted with IVb and a corresponding hydrochloride (VIb) was obtained. Phenothiazine reacts with VIb, forming VIIc (yield 34%). Alkoxides react with VIb, forming corresponding ethers. VIId and VIIe were obtained by the above reaction.

Card 5/10

Bicyclic Systems Based on 2,6-Lutidine. III

77375

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n	R	REACTION TIME (HR)	REACTION TEMPERATURE	YIELD (%)	BOILING POINT (PRESSURE IN MM)	MELTING POINT OF HYDROCHLORIDE
3	COCH ₃	4	On boiling	87	—	200—202°
3	COC ₂ H ₅	4	On boiling	58	—	170—172
3	COC ₆ H ₅	4	On boiling	80	—	189—191
3*		3	60—70°	59	183.5° (0.9)	179—181
3**		1	45—50	72	183 (1)	150—152

Card 6/10



(Continuation, and explanation of asterisks, on next card)

Bicyclic Systems Based on 2,6-Lutidine. III

77375

SOV/79-30-1-36/78

(table cont'd)

η	R	REACTION TIME (HR)	REACTION TEMPERATURE	YIELD (%)	BOILING POINT (PRESSURE IN MM)	MELTING POINT OF HYDROCHLORIDE
4	COCH ₃	4	On boiling	95	—	201—202
4	COC ₂ H ₅	4	On boiling	~100	—	494—496
4	COC ₆ H ₅	4	On boiling	87	—	194—195.5
4 *		2	60	67	200—201 (0.8)	137—139
4 **		2	60	60	184 (0.9)	152—154

* Was isolated in the form of dihydrochloride.

** Was isolated in the form of dihydrochloride monohydrate.

Card 7/10

Bicyclic Systems Based on 2,6-Lutidine. III

77375

SOV/79-30-1-36/78

The yields and properties of compounds are given below:

Compound	Yield (%)	bp (°C) (Pressure in mm)	mp (°C)
IIa	70	106-109/1	74-75
IIb	60	113-114/0.6	-
IIc	81	162-163/0.7	78-80
IId	72	183-185/0.2	-
IIe	75	140/0.8	68-70
IIf (1st fraction)	~30	101-103	-
IIf (2nd fraction)	56	260	-
IIg	27	-	138-139
IIh	78	124-126/0.5	77-79
IIi	83	157-159/0.55	97-99
IIj	90	148-150/0.4	100-102
IIk	43	-	140-142
IIl	81	67-67.5/3	-
IIm	64	55-56/0.8	-
IIIn	93	119-121/0.7	38-40

Card 8/10

Bicyclic Systems Based on 2,6-Lutidine. III

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SOV/79-30-1-36/78

(Continued from Card 8/10.)

The yield and properties of compounds are given below:

Compound	Yield (%)	bp (°C) (Pressure in mm)	mp (°C)
IIo	72	140-142/0.6	-
IIp	62	98-100/0.6	-
IIq	79	108/0.35	-
IIr	70	118-120/0.3	-
IIs	84	-	113-115
IIIa	77	157-159/0.7	-
IIIb	55	151-152/0.5	-
IIIc	77	171-172/1	63-65
IVa	65	107-109/0.5	-
IVb	70	135-137/1	-
VIa	75	217-219 (dec)	-
VIb	80	-	173-175
VIIa	41	-	234-236 (alc)
VIIb	52	215/0.8	-
VIIc	34	-	194-196
VIIId	-	-	163-165
Card 9/10	-	-	-

Bicyclic Systems Based on 2,6-Lutidine. III

77375

SOV/79-30-1-36/78

VIIe

(Continued from card 9/10.)

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176-177

There is 1 table; and 1 Soviet reference.

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SUBMITTED: January 21, 1959

Card 10/10

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77882

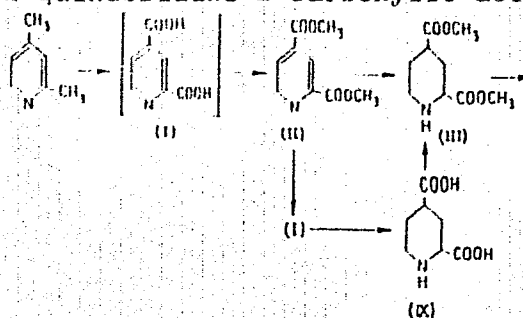
30V/79-30-2-33/78

AUTHORS: Yakhontov, L. N., Mastafanova, L. I., Rubtsov, M. V.

TITLE: Synthesis of 5-Substituted Quinolindine-2-Carboxylic Acid Based on 2,4-Lutidine

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol 30, Nr 2, pp 519-525 (USSR)

ABSTRACT: 5-Substituted quinuclidine-2-carboxylic acid was prepared.

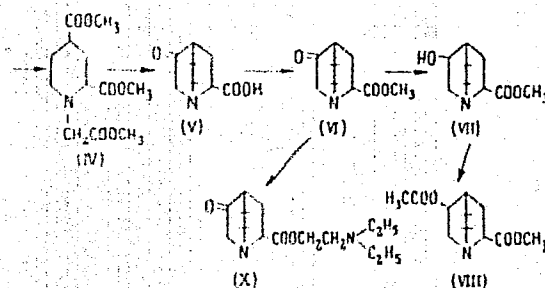


Card 1/5

Synthesis of 5-Substituted Quinolidine-
-2-Carboxylic Acid Based on 2,4-Lutidine

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SOV/79-30-2-33/78



The preparation of several compounds and some of their properties are given.

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Nr	Starting material	Obtained product	Yield in %	mp
1	Technical 2,4-lutidine + + H ₂ O + KMnO ₄	II	24.85	57.5-58.5
2	2,4-lutidine + formalin + + HNO ₃	II	56.5	57.5-58.5
3	2,4-pyridinedicarboxylic acid + HCl + hydrogenation over Pt	IX	89.7	224-226
4	dimethyl ester of 2,4-pyridi- nedicarboxylic acid + HCl + + methanol + Hydrogenation over Pt	III	84	151.5-152
5	corresponding 2,5-product was obtained in the same way		100	199.5-200

(Cont'd on Card 4/5)

Card 3/5

(Table cont'd)

77882 SOV/79-30-2-33/78

Nr	Starting material	Obtained product	Yield in %	mp
6	III + methyl bromoacetate + K_2CO_3	IV	56.2	bp 137-138 0.5 mm pr n_D^{20} 1.4717
7	anhydrous methanol + K + + IV	VI	61.2	bp 113-114 0.5 mm pr n_D^{20} 1.4848
8	VI + HCl	V	89.7	260 (dec)
9	diethylaminoethanol + sodium ethoxide + V	X	32.8	bp 162-165 2.5 mm pr n_D^{20} 1.4830

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(Cont'd on Card 5/6)

77882 SOV/79-30-2-33/78

Nr	Starting material	Obtained product	Yield in %	mp
10	VI + anhydrous methanol + + hydrogenation over Pt	VII	100	bp 135/0.3 mm n_D^{20} 1.5042
11	VII + acetic anhydride	VIII	50.6	bp 120/3 mm

There are 9 references, 1 Soviet, 3 German, 3 U.S., 1 U.K., 1 French. The 4 U.S. and U.K. references are: U.S. pat. 2456377 (1948); L. H. Sternbach, S. Kaiser, J. Am. Chem. Soc., 74, 2215 (1952); G. R. Clemco, T. P. Metcalfe, J. Chem. Soc., 1989 (1937); T. O. Solne, J. Am. Pharm. Ass., 33, 223 (1944).

ASSOCIATION: Ordzhonikidze All State-Scientific-Research Chemical-Pharmaceutical Institute (Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S. Ordzhonikidze)

SUBMITTED: February 2, 1959

Card 5/5

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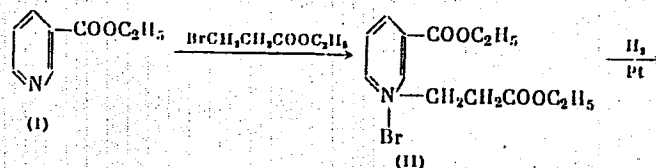
SOV/79-30-2-34/78

AUTHORS: Yanina, A. D., Rubtsov, M. V.

TITLE: Hoffmann Degradation of 1-Azabicyclo-(3,2,1)-Octanes.
II. Cleavage of 7-Methyl-1-Azabicyclo-(3,2,1)-Octane

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol 30, Nr 2,
pp 526-533 (USSR)

ABSTRACT: Quaternary base of 1-azabicyclo-(3,3,1)-nonane (VII),
on thermal decomposition, forms 1-methyl-3-allyl-
piperidine (VIII). The latter was used for the
preparation of the methiodide of 7-methyl-1-azabicy-
clo-(3,2,1)-octane (X).

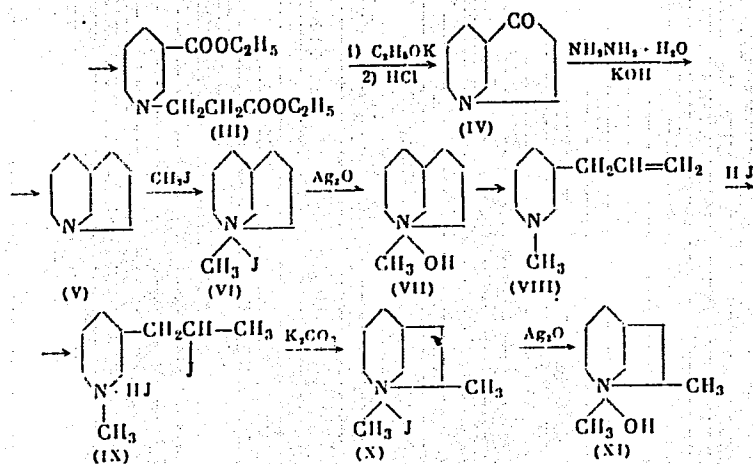


Card 1/9

(Figure continued on Card 2/9)

(Cont'd from Card 1/9)

777833 SOV/79-30-2-34/78

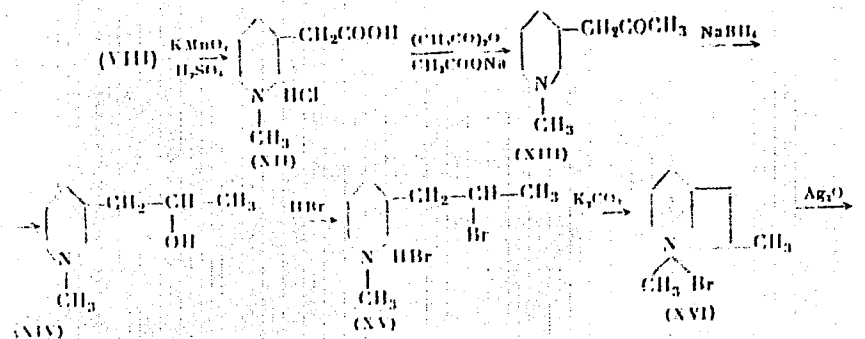


Card 2/9

Hoffmann Degradation of 1-Azabicyclo-
-(3,2,1)-Octanes. II. Cleavage of
7-Methyl-1-Azabicyclo-(3,2,1)-Octane

77883
SOV/79-30-2-34/78

Synthesis of X is based on the addition of HI to
VIII (according to Markownikow) with formation of
1-methyl-3-(β -iodopropyl)-piperidine (IX), and
cyclization to compound X. X was synthesized for the
final structure proof according to the scheme below:



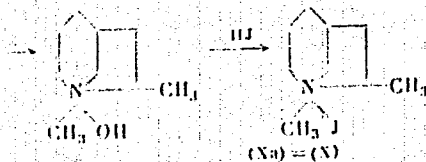
(Figure cont'd on Card 4/9)

Card 79

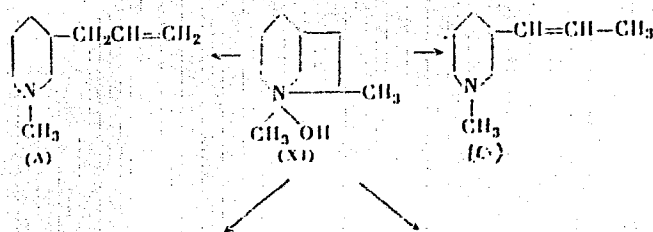
Hoffmann Degradation of 1-Azabicyclo-
-(3,2,1)-Octanes. II. Cleavage of
7-Methyl-1-Azabicyclo-(3,2,1)-Octane

77883

SOV/79-30-2-34/78



Theoretically, four possible compounds can be formed
by Hoffmann degradation of XI.



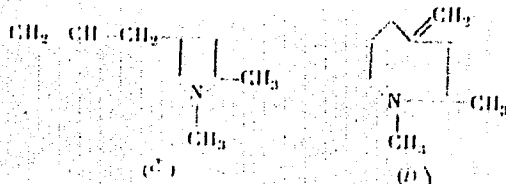
Card 4/9

(Figure cont'd on Card 5/9)

Hoffmann Degradation of 1-Azabicyclo-
-(3,2,1)-Octanes. II. Cleavage of
7-Methyl-1-Azabicyclo-(3,2,1)-Octane

77883

SOV/79-30-2-34/78



It was determined that only 1-methyl-3-allyl-piperi-
dine (A) is formed by degradation of XI.

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Card 6/9

77883 SOV/79-30-2-34/18

The Obtained Compounds and Their Properties

Nr	Starting Materials	Obtained product	Yield in %	bp/mm pr	n_D^{20}
1	ethyl ester of nicotinic acid + ethyl 2-bromopropionate + alcohol	III	71	123-125°/0.3	1.4581
2	III + K + anhydrous toluene + anhydrous alcohol	IV	48	95-100°/15	-
3	IV + KOH + hydrazine hydrate + glycerol	V	80	mp 84-86°	-
4	V + H ₂ O + AgOH	VIII	77	168-170°	1.4540
5	VIII + HI	IX	96	-	-

(Table cont'd)

77883 SOV/79-30-2-34/78

The Obtained Compounds and Their Properties

Nr	Starting Materials	Obtained product	Yield in %	bp/mm pr	n_D^{20}
6	IX + H ₂ O + ether + 50% solution of K ₂ CO ₃	X	69.7	mp 320°(dec)	-
7	VIII + H ₂ O + 10% H ₂ SO ₄ + KMnO ₄	ethyl-1-methylpiperidyl-3-acetate	72	115-116°/15	1.4540
8	ethyl-1-methylpiperidyl-3-acetate + HCl (1:1)	XII	90	mp 188-190°	-
9	XII + anhydrous CH ₃ COONa + acetic anhydride	XIII	33.5	95-97°/12	n_D^{18} 1.4632

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(Table cont'd)

77883 SOV/79-30-2-34/78

The Obtained Compounds and Their Properties

Nr.	Starting Materials	Obtained product	Yield in %	bp/mm pr	n_D^{20}
10	sodium borohydride + + CH_3OH + XIII	XIV	89	112-114°/1	n_D^{18} 1.4738

Four attempts were made at a Hoffmann degradation of compound XI: (1) H_2O + AgOH under normal pressure; (2) distillation under vacuum; (3) heating in the presence of alkali; (4) heating in the presence of water, under pressure. In all cases, only VIII was obtained (corresponding in 80, 66, 84.5, and 64% yields) with bp 168-170°, $n_D^{20.5}$ 1.4540. There are 4 references, 1 Soviet, 3 U.S. The 3 U.S. references are: L. H. Sternbach, S. Kaiser, J. Am. Chem. Soc., 74, 2215 (1952); N. J. Leonard, E. Barthel, Jr.,

Card 6/9

Hoffmann Degradation of 1-Azabicyclo-
-(3,2,1)-Octanes. II. Cleavage of
7-Methyl-1-Azabicyclo-(3,2,1)-Octane

77883

SOV/79-30-2-34/78

ibid, 71, 3098 (1949); A. Bugger, C. R. Walter,
ibid, 72, 1988 (1950).

ASSOCIATION:

Ordzhonikidze All State Scientific-Research Chemical-
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SUBMITTED:

February 21, 1959

Card 9/9

RUBTSOV, M.V.

Synthesis of racemic N-acetylhomomeroquinene. Zhur.ob.khim.
30 no.5:1498-1507 My '60. (MIRA 13:5)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S.Ordzhonikidze.
(Homomeroquinene)

YAKHONTOV, L.N.; RUBTSOV, M.V.

Synthesis of 3(α -diethylaminoethyl)4-methylpyridine. Zhur.ob.
khim. 30 no.5:1507-1515 My '60. (MIRA 13:5)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S.Ordzhonikidze.
(Pyridine)

MIKHLINA, Ye.Ye.; VOROB'YEVA, V.Ya.; RUBTSOV, M.V.

Synthesis of 3- and 4-hydroxypiperidine derivatives. Zhur.ob.
khim. 30 no.6:1885-1893 Je '60. (MIRA 13:6)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevti-
cheskiy institut imeni S. Ordzhonikidze.
(Piperidine)

YANINA, A.D.; RUBTSOV, M.V.

Hofmann cleavage of 1-azabicyclo [3.3.1]octanes. Part 3: Hofmann
cleavage of a quaternary base of 2-methyl-1-azabicyclo[3.2.1]octane.
Zhur.ob.khim. 30 no.8:2544-2550Ag '60. (MIRA 13:8)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S.Ordzhonikidze.
(Azabicyclooctane)

MIKHLINA, Ye.Ye.; RUBTSOV, M.V.

New steps toward the synthesis of 3-quinuclidineacetic acid. Zhur;
ob. khim. 30 no.9:2970-2977 S '60. (MIRA 13:9)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S. Ordzhonikidze.
(Quinuclidineacetic acid)

MIKHILINA, Ye.Ye.; VOROB'YEVA, V. Ya.; RUETSOV, M.V.

Synthesis of polymethylene-bis-quinoccludinium halides. Zhur.
ob.khim. 31 no.8:2609-2613 Ag '61. (MIRA 14:8)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S. Ordzhonikidze.
(Quinoccludinium compounds)
(Polymethylene compounds)

YAKHONTOV, L.N.; KRASNOKUTSKAYA, D.M.; RUBTSOV, M.V.

Synthesis and some conversions of 1-phenyl-1-oxy-2-methoxy-
methylcyclohexane. Zhur.ob.khim. 31 no.10:3190-3197 0 '61.
(MIRA 14:10)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S.Ordzhonikidze.

(Benzene)

NIKITSKAYA, Ye.S.; USOVSKAYA, V.S.; RUBTSOV, M.V.

Bicyclic systems based on 2, 6-lutidine. Part 5: Biquaternary salts of α, ω -bis[9-methyl-3, 9-diazabicyclo (3, 3, 1)-nonano-3]-alkanes. Zhur.ob.khim. 31 no.10:3202-3205 0 '61. (MIRA 14:10)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut imeni S.Ordzhonikidze.
(Lutidine) (Paraffins)

MIKHLINA, Ye.Ye.; RUBTSOV, M.V.; VOROB'YEVA, V.Ya.

Synthesis of quinuclidine-2, 3-dicarboxylic acid. Zhur.ob.khim.
31 no.10:3251-3255 0 '61. (MIRA 14:10)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-farmatsevticheskiy
institut imeni S.Ordzhonikidze.
(Quinuclidinecarboxylic acid)

YANINA, A.D.; RUBTSOV, M.V.

Hofmann degradation of 1-azabicyclo(3,2,1)octanes.
Part 5: Hofmann degradation of 6-methyl-1-azabicyclo(3,2,1)
octane. Zhur.ob.khim. 32 no.10:3151-3158 0 '62. (MIRA 15:11)

1. Vsesoyuznyy nauchno-issledovatel'skiy khimiko-
farmatsevticheskiy institut imeni S. Ordzhonikidze.
(Azabicyclooctane) (Degradation)

RUBTSOV, M. V.

AUTHORS: Nikitskaya, Ye. S., Rubtsov, M. V. 79-11-46/56

TITLE: Synthesis of Bicyclic Systems Starting From 2,6-Lutidine.
Synthesis of 9-Methyl-2-Oxy-9-Azabicyclo (3,3,1)-Nonanes
(Azobicyclic)
(Sintez bitsiklicheskikh sistem, iskhodya iz 2,6-lutidina)
(Sintez 9-Metil- 2- oksi - 9 - azabitsiklo (3,3,1) - nonana).

PERIODICAL: Zhurnal Obshchey Khimii, 1957, Vol. 27, Nr 11,
pp. 3133-3136 (USSR)

ABSTRACT: The investigation of the azobicyclic compounds of the octane series (quinuclidine, tropane) showed that they are of great interest as raw products for the synthesis of remedies. Thus compounds with curative, ganglion-blocking, spastmatic, mydriatic and other properties were discovered among the tropine derivatives. It was of interest to investigate the bicyclic systems close to the tropane series. Thus the authors synthesized 9-methyl-2-oxy-9-azobicyclo- (3,3,1)-nonane by starting from the ethyl ester of 6-methylpicolinic acid (obtained from 2,6-lutidine). (See the process of reaction). The initial, intermediate and final products are as follows:

Card 1/2 the ethyl ester of 6-methylpicolinic acid, the product of

Synthesis of Bicyclic Systems Starting From 2,6-Lutidine. 79-11-46/56
Synthesis of 9-Methyl-2-Oxy-9-Azabicyclo (3,3,1)-Nonanes (Azobicyclic)

its condensation with chloral, 2-carboxy-6- (β -carboxyvinyl)-pyridine, 2-carbethoxy-6- (β -carbethoxyethyl)-piperidine, 9-methyl-2-keto-9-azobicyclo (3,3,1)-nonane which on reduction with aluminumhydrate of lithium is converted to 9-methyl-2-oxy-9-azobicyclo (3,3,1)-nonane.
There are 3 references, 1 of which is Slavic.

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1. Cyclic compounds - Synthesis

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